(12) INTERNATIONAL APPLICATION PUBLISHED UNDER THE PATENT COOPERATION TREATY (PCT)

(19) World Intellectual Property Organization
International Bureau

(43) International Publication Date
26 January 2006 (26.01.2006)

PCT

(10) International Publication Number
WO 2006/009331 A1

(51) **International Patent Classification**[7]**:** B29C 71/00

(21) **International Application Number:** PCT/KR2004/001813

(22) **International Filing Date:** 21 July 2004 (21 07 2004)

(25) **Filing Language:** English

(26) **Publication Language:** English

(71) **Applicant** *(for all designated States except US):* **NCT, CO., LTD.** [KR/KR], 552 27, Kajwa Dong, Seo Ku, Incheon 404 250 (KR)

(72) **Inventor; and**
(75) **Inventor/Applicant** *(for US only):* **ALEKSANDR, Tsoi** [RU/KR], 552 27, Kajwa Dong, Seo Ku, Incheon 404 250 (KR)

(74) **Agents: JUNG, Seung-Hun** et al , Dabong Tower Building, 10th Fl „ 890 12,Daechi/dong, Kangnam gu, Seoul 135 280 (KR)

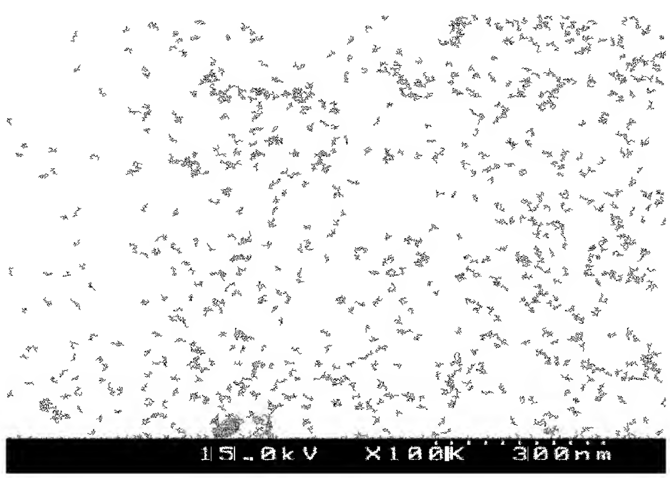
(81) **Designated States** *(unless otherwise indicated for e\ery kind of national protection a\aVable):* AE, AG, AL, AM, AT, AU, AZ, BA, BB, BG, BR, BW, BY, BZ, CA, CH, CN, CO, CR, CU, CZ, DE, DK, DM, DZ, EC, EE, EG, ES, FI, GB, GD, GE, GH, GM, HR, HU, ID, IL, IN, IS, JP, KE, KG, KP, KR, KZ, LC, LK, LR, LS, LT, LU, LV, MA, MD, MG, MK, MN, MW, MX, MZ, NA, NI, NO, NZ, OM, PG, PH, PL, PT, RO, RU, SC, SD, SE, SG, SK, SL, SY, TJ, TM, TN, TR, TT, TZ, UA, UG, US, UZ, VC, VN, YU, ZA, ZM, ZW

(84) **Designated States** *(unless otherwise indicated for e\ery kind of regional protection a\ailable):* ARIPO (BW, GH, GM, KE, LS, MW, MZ, NA, SD, SL, SZ, TZ, UG, ZM, ZW), Eurasian (AM, AZ, BY, KG, KZ, MD, RU, TJ, TM), European (AT, BE, BG, CH, CY, CZ, DE, DK, EE, ES, FI, FR, GB, GR, HU, IE, IT, LU, MC, NL, PL, PT, RO, SE, SI, SK, TR), OAPI (BF, **BJ**, CF, CG, CI, CM, GA, GN, GQ, GW, ML, MR, NE, SN, TD, TG)

Published:
— *with international search report*

For two letter codes and other abbreλiations refer to the Guidance Notes on Codes and Abbreλiations appearing at the beginning of each regular issue of the PCT Gazette

(54) **Title:** METHOD FOR PRODUCTION OF SPIRAL SHAPED CARBON COATED WITH NANO CRYSTALLINE STRUCTURED CARBON LAYER AND INFRARED EMITTER COMPRISING SPIRAL SHAPED CARBON



(57) **Abstract:** The present invention relates to a method for making a spiral shaped carbon material as a heating element used for an infrared emitter An initial carbon material formed of carbon fibers is wound onto a mandrel in the form of spiral The carbon material is saturated with resin solution The carbon mateπal is heated by applying an electric current via the electric contacts in an inert or reducing atmosphere After finishing this first heating process, the carbon material is cooled and taken off the quartz tube The strengthened spiral carbon material is re heated by applying the electπc current in gaseous hydrocarbon atmosphere As a result, a coat of nano crystalline structure carbon is formed on a surface of the carbon mateπal The spiral shaped carbon material with nano crystalline structured carbon coating layer shows low thermal inertness and maximal radiating ability in comparison with other type of heating elements, including the elements made of usual carbon fiber as well

WO 2006/009331 A1

Description

METHOD FOR PRODUCTION OF SPIRAL-SHAPED CARBON COATED WITH NANO-CRYSTALLINE STRUCTURED CARBON LAYER AND INFRARED EMITTER COMPRISING SPIRAL-SHAPED CARBON

[1] Technical Field

[2] The present invention concerns to the method for production of a carbon heating element used for an infrared emitter, more specifically a spiral-shaped carbon coated with a nano-crystalline structured carbon layer by precipitation from gaseous phase. Furthermore, the present invention concerns to an infrared emitter comprising the spiral-shaped carbon as an infrared electro-heating element.

[3] Background Art

[4] It is known the method from USA Patent № 6,464,918 from 15 Oct. 2002. In this Patent the method for making spiral-shaped heating elements of carbon fiber is described. Carbon fiber heating element is saturated with thermo-plastic polymeric material. The method includes the following stages: heating an initial material up to softening temperature of strengthening compound; winding of heated initial material onto mandrel so as to make a spiral; and keeping the spiral shape by removing of strengthening compound wherein removing of strengthening compound includes annealing spiral at the temperature and in the atmosphere which transforms strengthening material into volatile material.

[5] This method allows to obtain spiral-shaped carbon heating element having better radiating ability and low inertia of heating velocity because of large carbon spiral radiating surface. In this case the maximum surface density of electric energy can mount to 195KW/m^2. However, in the case of the increasing surface density of electric energy the carbon material overheating and its destruction take place. At maximum surface density of electric energy, the luminescence temperature mounls to 1,200 ℃ through 1,300 ℃ . These characteristics are better in comparison with metal heating elements such as nickel-chromium, tungsten, and other alloys, but not the best.

[6] Meanwhile, it is known that nano-crystalline materials on the base of carbon and other elements have unique physical, electrical, mechanical and electronic properties. High mechanical strength and thermo-resistance in combination with their electro-conductivity give a good possibility to use them for production of resistive heating

elements such as light sources, electrodes for electrochemical processes and so on. The pores in the nano-crystalline material have nano-size that gives possibility to obtain special optical characteristics: mixing of absorption and omission stripes; appearance of additional stripes; possibility of pore-resonance because nano-meter size is less than light wave length. It gives possibility to make, for example, heating resistive electric element in which visible light quantum transforms into infra-red radiation. Thus, it gives possibility to make high-temperature heater which completely converts electric energy into heat. When the pores of nano-crystalline material are filled up with kations of corresponding elements, for example, rare-earth ones, An inverse effect is possible where the heat photon transforms into visible light quantum, i.e. it gives possibility to make very high-effective light sources.

[7] There are many methods for obtaining nano-crystalline material (A.V.Elelskiy, Carbon nano-tubes. Physical science progress, V.167. #9, pp.945-972. 1997): thermal dispersion in electric arc; laser dispersion; electrolytic synthesis; catalytic acetylene cracking and so on. All methods can be conditionally divided into catalytic and non-catalytic ones. Catalytic methods give possibility to obtain nano-carbon material at low temperature (under 1,200 ^{0}C) in the presence of catalyst, but only in powder form. Non-catalytic methods need higher temperature, but in this case process productivity is very low and final product is obtained in the form of fragile coat (thin coating) on cooled support.

[8] Method of resistive heating is described in Japan Patent (JN°2000272913 from 25.03.2002), where carbon granules are used as dispersed material; electric current is passed through the granules and the temperature mounts up to 3,400 ^{0}C . Carbon granules are in the medium of inert gas. Carbon vapor precipitates onto rotating water-cooled support and the coat of nano-crystalline material forms on the support's surface at temperature of 2,500 ^{0}C . However, this method doesn't let to obtain thickness-uniformed coat with low adhesion to support.

[9] On the other side, there are methods to bring pyrolytic coat onto carbon fiber with gas-phase process, for example, pyrolysis of hydrocarbons (methane, propane, butane, acetylene and so on). As a rule, the process is carried out in running furnace where hydrocarbon gas is heated and it decomposes on to carbon and other components; the temperature is not over 1,300 ^{0}C . Pyrolytic carbon structure is close to graphite structure and has higher density and electro-conductivity in comparison with carbon fiber.

[10] The goods of composition material on the base of carbon fiber are made for using

at high temperatures in oxidizing mediums. Besides, composition materials on the base of carbon fiber have high electro-conductivity and can be used for producing heater and illuminator. It is known the method to obtain composition material on the base of carbon fiber and silicon-carbide (Russian Patent RU 205 8964 C1 on 27.04.1996), where carbon semi product is made from two layeis: the main layer has carbon fiber with low reactive ability to silicon and the surface layer has carbon fiber with high-limited reactive ability. Semi product can be made by dint of selection from carbon fiber with different densities or by making half-finished product surface layer from carbon fiber with barrier coating from pyro-carbon and/or silicon-carbide. At such conditions normal crystalline structure with higher density forms, but nano-crystalline structure doesn't form; the process velocity is very low and can't be used in industrial production.

[11] Furthermore, there is the method from Russian Patent JN°2149215 C1 from 20.05.2000, where the method for obtaining pyro-carbon layeis is described. According to the Russian Patent, obtaining carbon material is carried out by precipitation of pyrolytic carbon layer from gas phase onto various material surfaces. Precipitated pyrolytic carbon layer has high crystalline structure regulation degree. Pre cipitation process is carried out onto heated surface under atmosphere pressure and temperature range of 950 ^{0}C through 1,350 ^{0}C from steam-gas mixture of carbon tetra-chloride and hydrogen in 1 : (5 through 50) ratio. This invention lets increase life-time of graphite goods on the base of carbon composition material and decrease their cost price. When basing the pyrolytic carbon precipitation process parameteis, it is indicated that increasing carbon tetra-chloride : hydrogen ratio more than 1 : 5 leads to homogeneous reaction, and as a result soot (carbon-black) sediment precipitates within reactor and on the reactor's walls. Decreasing carbon tetra-chloride : hydrogen ratio less than 1 : 50 leads to lowering obtained pyrolytic-carbon crystallization degree and precipitation process efficiency. When the precipitation process is carried out at temperature below 950 ^{0}C , pyro-carbon formation doesn't take place and the sediment has soot structure. Heating of the support up to temperature above 1,350 ^{0}C is also undesirable because of decreasing structure regulation degree of pyro-carbon sediment and its density, and as well precipitation velocity decreasing.

[12] Maximum pyro-carbon density, achieved in this method, is 2.21g/cm ^{3} , and maximum pyro-carbon precipitation velocity is 1,250 m m/h at volume expenditure of steam-gas mixture 0.5m ^{3}/h or 8.3L/min. This method can be used for brining protective coating the composition material, but can't be used for making composition

material and goods, because precipitation velocity is very low and doesn't ensure coating uniformity on sediment thickness. In this method the structure is very close to pure graphite structure, but not is nano-crystalline.

[13] Disclosure of the Invention

[14] Therefore, the present invention has been made in view of the above problems. The purpose of the present invention is to provide the method for obtaining a carbon on the base of carbon fibeis with pyrolytic coat of nano-crystalline structured carbon in the form of spiral. According to the present invention, precipitation process productivity is at least in ten times more in comparison with known methods.

[15] The invented method allows the production of a spiral-shaped carbon material coated with a nano-crystalline structured carbon layer, said method comprising the steps of: (a) winding a carbon material formed of a plurality of carbon fibeis onto a mandrel in the form of a spiral; (b) fitting both ends of the carbon material with contacts for electrical connection; (c) saturating the carbon material with resin solution; (d) heating the carbon material wound onto the mandrel by means of applying an electric current on the carbon material via the contacts thereof in an inert or reducing atmosphere; (e) taking the carbon material off the mandrel; and (f) re-heating the carbon material by means of applying an electric current on the carbon material via the contacts thereof in a medium of gaseous hydrocarbon.

[16] According to the preferred method of the present invention, heating of the carbon material is carried out by means of a resistive heating wherein an electric current is applied on the carbon material. Especially, during the step of (d) of the present method, the carbon material is heated up to a temperature when resin pyrolysis starts and during time that is necessary for fixing a form of spiral. In this heating process, the temperature should be not more than the melting point of the mandrel. When a quartz tube is used as the mandrel, the electric current is applied on the carbon material so that a surface density of electric energy is not less than $20W/cm^2$ and not more than $50W/cm^2$ in re-count to surface area of the carbon material.

[17] As far as the step of (f) is concerned, the carbon material is re-heated in a temperature range of 1,400 ^{0}C through 2,500 ^{0}C on a surface of the carbon material for the purpose of the stable formation of a nano-crystalline structured carbon coating, and the work efficiency. Accordingly, in the step of (f), the electric current is applied so that a surface density of electric energy is not less than $50W/cm^2$ and not more than $200W/cm^2$ in re-count to surface area of the carbon material. Furthermore, in the step of (f), the carbon material is heated in a furnace through which the gaseous hy-

drocarbon is blown at gas expenditure of 0.015L/min- cm^2 through 0.15L/min- cm^2.

[18] Furthermore, the other purpose of the present invention is to provide the infrared emitter comprising the spiral-shaped carbon material coated with a nano-crystalline structured carbon layer as a heating element. The infrared emitter according to the present invention distinguishes its low heat inertia, and provides maximum limited radiating ability at any temperatures.

[19] Brief Description of the Drawings

[20] The above and other objects, features and other advantages of the present invention will be more clearly understood from the following detailed description taken in conjunction with the accompanying drawings, in which:

[21] Fig. 1 shows the X-ray diffraction diagram for the carbon material produced by the method according to the present invention;

[22] Fig. 2 shows the X-ray diffraction diagram for initial carbon fibers; and

[23] Fig. 3 shows the electronic-microscope picture of the surface of the carbon material coated with a nano-crystalline structured carbon layer.

[24] Best Mode for Carrying Out the Invention

[25] Now, preferred embodiment of a method for production of a spiral-shaped carbon material according to the present invention will be described in detail.

[26] Fiist, as an initial material, it is prepared carbon fibeis of 4 parallel viscose threads with lOOtex linear density. Next, the carbon fibeis are wound onto a mandrel by a winding device. The mandrel is made of a quartz tube, and has a form of bar with 5mm diameter. The carbon fibeis are wound in the form of a spiral-shaped ribbon with step of 2 per lcm and length of 30cm. The material formed of the carbon fibeis has straight ends of twisted threads. The ends are fitted with contacts for electrical connection. The contacts are made of aluminum or copper sticking foil.

[27] After preparing the carbon material for winding onto the mandrel in the form of spiral, the carbon material is saturated with polymer phenol -formaldehyde resin solution together with the mandrel . Although any resin solution is available, phenol-formaldehyde resin is preferable because it contains much carbon. After drying the carbon material, the electric contacts of the carbon material are fixed into a special carrier with electric terminals .

[28] Next, the special carrier is placed into a reactor. The reactor is preferably is provided with a quartz tube having a large inside diameter. The special carrier where the carbon ribbon is fixed into is placed inside of the reactor. And then, an electric current such as an alternating or direct current is applied on the carbon material via the

electric contacts thereof. Furthermore, applying the electric current is performed in an inert or reducing atmosphere. Preferably, any gaseous hydrocarbon containing carbon and hydrogen, such as methane and acetylene, is blown through the inside of the reactor during applying the electric current on the carbon material. In this case, for safety, the gas is burnt on the outlet of the reactor.

[29] While applying the electric current on the carbon material in this manner, the carbon material warms up and a pyrolysis reaction of the phenol-formaldehyde resin takes place on a surface of the carbon material. As a result of the pyrolysis reaction, the carbon coating is formed on the surface of the carbon material. Preferably, heating of the carbon material is performed up to temperature when the phenol-formaldehyde resin pyrolysis reaction starls and is kept during time that is necessary for fixing the form of spiral by pyrolytic carbon.

[30] In this heating process (hereinafter, referred to 'first heating process'), when the electric current is applied on the carbon material, the surface density of electric energy must be not less than $20W/cm^2$ on the surface of the carbon material. The area of the carbon material can be calculated from the thread length and the thread width on the mandrel. In case the thread length is 126cm and the thread width is 3mm, the area is to be $38cm^2$. Initial resistance is 158 ohm. At 380 voltages, the surface density of electric energy is $24W/cm^2$ and the initial power is 920W. After phenol-formaldehyde de-composition, the conductivity increases and current grows correspondingly from 2.5A to 3.0A. Further, because of methane pyrolysis, the current increases gradually up to 5.0A that corresponds to $50W/cm^2$ in re-count on the surface density of electric energy.

[31] In this fiist heating process, the surface density of electric energy must be not less than $20W/cm^2$ and not more than $50W/cm^2$. If the surface density of electric energy is less than $20W/cm^2$, the pyrolytic carbon is not enough for keeping the spiral form. Moreover, because carbon fibeis of the initial material contain the amorphous graphite-like componenls as well as the graphite-crystalline components, it is necessary to transform the amorphous componenls into the graphite-crystalline componenls. Accordingly, the carbon material should be heated up to at least the temperature necessary for crystallization of the amorphous componenls. However, when the surface density of electric energy is less than $20W/cm^2$, the temperature on the surface of the carbon material would not go up to the temperature necessary for crystallization of the amorphous componenls. Furthermore, if the surface density of electric energy is more than $50W/cm^2$, the mandrel made of quartz tube starls to melt (i.e. in this case the temperature exceeds 1,300 ^0C that is a soften temperature for

quartz glass).

[32] By means of the fiist heating process, it is formed into the strengthened spiral of the carbon material. After cooling the reactor with the strengthened spiral of the carbon material, the carbon material is taken out from pyrolysis zone and taken off the mandrel. And t hen , the spiral -shaped carbon material is fixed into another special carrier with the electric terminals and placed again into the reactor.

[33] The spiral-shaped carbon material without the mandrel is heated by means of applying the electric current on the carbon material via the contacts thereof and the carrier. Further, any gaseous hydrocarbon such as methane is passed through the reactor. Hereinafter, this heating process is referred to second heating process. This second heating process is performed at 380 voltages and fiist current of 4.8A (that corresponds to $48W/cm^2$ of the surface density of electric energy) up to 6.5 A (that corresponds to $65W/cm^2$ of the surface density of electric energy). In this period, the temperature is above 2,200 ^0C . During the second heating process, the pyrolysis reaction takes place again .

[34] The specific expenditure of the gaseous hydrocarbon plays a large role in the pyrolysis process. When gas expenditure is less than 0.015 $L/min\text{-}cm^2$ (gas amount per minute and unit area of the surface of the carbon material), the pyrolysis process proceeds irregularly along the length of the spiral- shaped carbon material and a carbon coating is not identical on thickness. On the other hand, when gas expenditure is more than 0.15 $L/min\text{-}cm^2$, one of ends of the carbon material cools thus forming non-identical nano-crystalline carbon coat on the spiral-shaped carbon material.

[35] During the second heating process, the given surface density of electric energy must be not less than $50W/cm^2$ and not more than $200W/cm^2$. When the energy density is less than $50W/cm^2$, the precipitation velocity is very low. Moreover, in order to form a nano-crystalline structured carbon, the temperature on the surface of the carbon sh ould be not less than 1,400 ^0C that approximately corresponds to the surface density of electric energy of $50W/cm^2$. On the other hand, when the energy density is more than $200W/cm^2$, the pyrolysis hydrocarbon decomposition it takes place not only on the carbon material but also within the furnace made of quartz tube around the carbon ribbon. When the pyrolysis hydrocarbon decomposition takes place far from the carbon material, the amount of hydrocarbon around the carbon material may be decreased. As a result, the efficiency of forming a nano-crystalline structured carbon coat may be deteriorated.

[36] Substantiation of selected parameteis for the second heating process is shown in

Table 1. Herein, 'density' means the density of the nano-crystalline structured carbon coat formed by the method according to the present invention.

[37] Table 1

[38]

№	Hydrocarbon gas expenditure (L/min ·cm^2)	surface density of electric energy (W/cm²)	Precipitation velocity (μm/min)	Density (g/cm³)
1	0.015	50	130	2.55
2	0.06	70	250	2.63
3	0.15	100	400	2.60
4	0.06	45	40	2.28
5	0.09	110	150	2.45
6	0.015	70	80	2.64
7	0.165	70	120	2.52

[39] By means of the second heating process, the nano-crystalline structured carbon coating layer is formed on the surface of the carbon material. To prove the formation of the nano-crystalline structured carbon coating layer, we adduce the next data: radio-graphic diagram for the carbon material produced by the method according to the present invention as shown in Fig. 1; radio-graphic diagram for initial carbon fiber as shown in Fig. 2 ; and electronic-microscope picture of the surface of the carbon material coated with a nano-crystalline structured carbon layer as shown in Fig. 3.

[40] As known, graphite crystals are endless nels of hexagonal rings. The atoms in the nels are connected with each otheis by covalent bonds; the shortest distance between atoms is 1.42 Å ; the number of nearest neighbors in the net is three (3) . The nels dispose themselves flaky one under other and the distance between layers is 3.35 Å . Thus, the most characteristic inter-layer distance for graphite is d=3.35 Å or 0.345nm. Characteristic inter-layer distance for nano-crystalline structure d (002) =0.340 through 0.350nm. Diamond structure can be presented as two HCC(hexahedron center cubic) lattices with displacement on 1/4 of body diagonal. The most strong reflection from layer family (111) is at d=0.357nm. Thus, nano-structure as is in the middle between diamond and graphite.

[41] Fig. 1 and 2 show two radio-graphic diagrams in comparison. As seen from diagrams shown in Fig. 1 , the initial carbon fiber has exceptionally graphite structure with low crystallization degree and inter-plane distance for 002 plane is 0.335nm; whereas for the carbon material obtained with the method according to the present invention , as shown in Fig. 2, inter-plane distance for 002 plane is 0.348 through 0.350nm. It proves the aforesaid.

[42] Fig. 3 shows an electronic-microscope picture of the surface of the carbon material obtained from the method according to the present invention . As seen from Fig. 3 , formed carbon coat has roll structure, i.e. rolled many-layeis coat around carbon filament . Coat's surface is scaly structure of irregular polygons. After oxidation such polygons have turned up borders. Polygons' size is within some nano-meteis.

[43] Electro-conductivity calculations showed that nano-crystalline coat conductivity was in 5 through 7 times higher than for carbon fiber in re-calculation for identical density.

[44] The present method gives possibility to make spiral heating elements with a coat of nano-crystalline structured carbon. Thanks to such nano- crystalline structure , the thermo-radiating characteristics of heating element grow up to maxim um possibly. For proof , Table 2 where the test resulls for various electric heateis placed into water is shown.

[45] For comparison , we used the following heateis: metal nickel-chromium heater is soldered into U-quartz tube under vacuum; halogen heater; standard carbon heater; and carbon heater with nano-crystalline structure. Size, shape and electric capacity for all radiators are the same. Electric capacity is 1 , 00OW, water volume is 31iter, first water temperature is 17 ℃ and the finish temperature is 70 ℃ . Theoretical calculation shows that in the ideal case for 100% transformation of electric energy into heat it is necessary to spend 1.24W for heating 1 liter of water per 1 degree.

[46] Table 2

[47]

Heater	Heating time up to 70 ℃	Spent capacity (W/liter.degree)	Efficiency (%)
Nickel-chromium	12min 25sec	1.30	95.2
Halogen	12min 45sec	1.34	92.0
Standard carbon	12min 8sec	1.27	97.5
Nano-crystalline	11min 45sec	1.24	100.0

[48] Thus, nano-crystalline carbon heater has maximal caloric ability in comparison with another kind of electric heateis. It is important to note that during work the nano-crystalline carbon heater absolutely doesn't radiate luminescence, while the otheis do.

[49] Although the preferred embodiments of the present invention have been disclosed for illustrative purposes, these skilled in the art will appreciate that various modifications, additions and substitutions are possible, without departing from the scope and spirit of the invention as disclosed in the accompanying claims.

Claims

[1] 1. The method for production of a spiral-shaped carbon material coated with a nano-crystalline structured carbon layer, said method comprising the steps of:
(a) winding a carbon material formed of a plurality of carbon fibeis onto a mandrel in the form of a spiral;
(b) fitting both ends of the carbon material with contacts for electrical connection;
(c) saturating the carbon material with resin solution;
(d) heating the carbon material wound onto the mandrel by means of applying an electric current on the carbon material via the contacts thereof in an inert or reducing atmosphere;
(e) taking the carbon material off the mandrel; and
(f) re-heating the carbon material by means of applying an electric current on the carbon material via the contacts thereof in a medium of gaseous hydrocarbon.

[2] 2. The method according to claim 1, wherein the mandrel is formed of a quartz grass.

[3] 3. The method according to claim 1, wherein the resin solution is phenol-formaldehyde resin solution.

[4] 4. The method according to claim 1, wherein in the step of (d), the carbon mater ial is heated up to a temperature when resin pyrolysis starts and is kept during time that is necessary for fixing a form of spiral.

[5] 5. The method according to claim 4, wherein in the step of (d), the temperature is not more than the melting point of the mandrel.

[6] 6. The method according to claim 2, wherein in the step of (d), the electric current is applied so that a surface density of electric energy is not less than $20W/cm^2$ and not more than $50W/cm^2$ in re-count to surface area of the carbon material.

[7] 7. The method according to claim 1, wherein in the step of (d), the carbon material is heated in a medium of gaseous hydrocarbon.

[8] 8. The method according to claim 1, wherein in the step of (f), the carbon material is re-heated in a temperature range of 1,400 ^0C through 2,500 ^0C on a surface of the carbon material.

[9] 9. The method according to claim 1, wherein in the step of (f), the electric current is applied so that a surface density of electric energy is not less than

$50W/cm^2$ and not more than $200W/cm^2$ in re-count to surface area of the carbon material.

[10] 10. The method according to claim 1, wherein in the step of (f), the carbon material is heated in a reactor through which the gaseous hydrocarbon is blown at gas expenditure of $0.015L/min\text{-}cm^2$ through $0.15L/min\text{-}cm^2$.

[11] 11. A infrared emitter comprising the spiral-shaped carbon material produced by the method described in any one of claims 1 through 10 as a heating element.

[12] 12. A method for production of a spiral-shaped carbon material coated with a nano-crystalline structured carbon layer, said method comprising the steps of:
(a) winding a carbon material formed of a plurality of carbon fibeis onto a mandrel in the form of a spiral, the mandrel being formed of a quartz glass;
(b) fitting both ends of the carbon material with contacts for electrical connection;
(c) saturating the carbon material with resin solution;
(d) heating the carbon material wound onto the mandrel by means of applying an electric current on the carbon material via the contacts thereof in an inert or reducing atmosphere, the electric current being applied so that a surface density of electric energy is not less than $20W/cm^2$ and not more than $50W/cm^2$ in re-count to surface area of the carbon material;
(e) taking the carbon material off the mandrel; and
(f) re-heating the carbon material by means of applying an electric current on the carbon material via the contacts thereof in a medium of gaseous hydrocarbon, the electric current being applied so that a surface density of electric energy is not less than $50W/cm^2$ and not more than $200W/cm^2$ in re-count to surface area of the carbon material.

[13] 13. The method according to claim 12, wherein the resin solution is phenol-formaldehyde resin solution.

[14] 14. The method according to claim 12, wherein in the step of (d), the carbon material is heated in a medium of gaseous hydrocarbon.

[15] 15. The method according to claim 12, wherein in the step of (f), the carbon material is heated in a reactor through which the gaseous hydrocarbon is blown at gas expenditure of $0.015L/min\text{-}cm^2$ through $0.15L/min\text{-}cm^2$.

[16] 16. A infrared emitter comprising the spiral-shaped carbon material produced by the method described in any one of claims 12 through 15 as a heating element.

FIG. 1

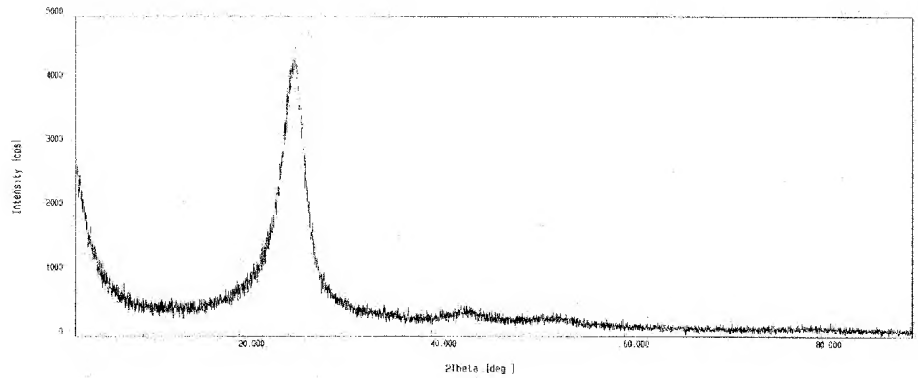

5000
4000
3000
2000
1000
0
Intensity [cps]
20.000
40.000
60.000
80.000
2Theta [deg]

FIG. 2

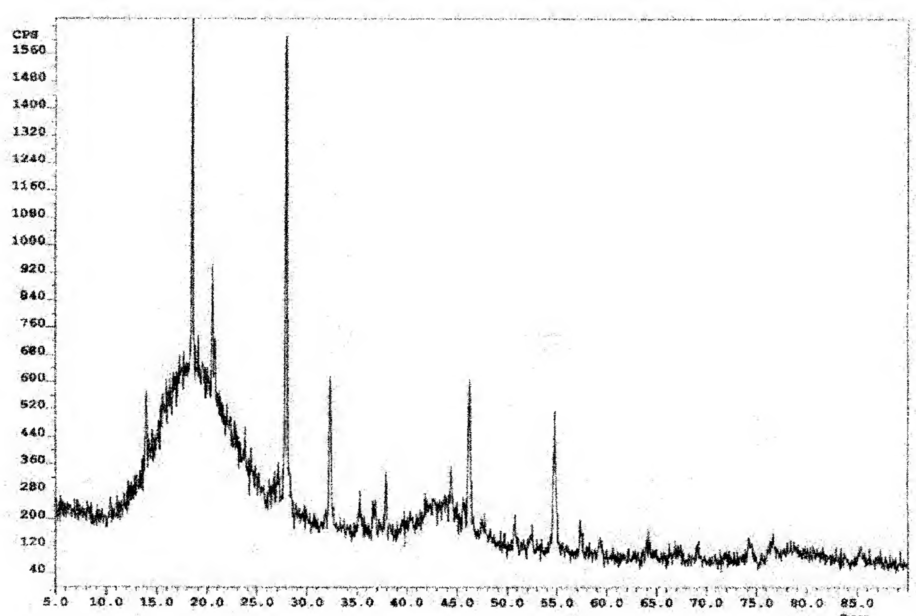
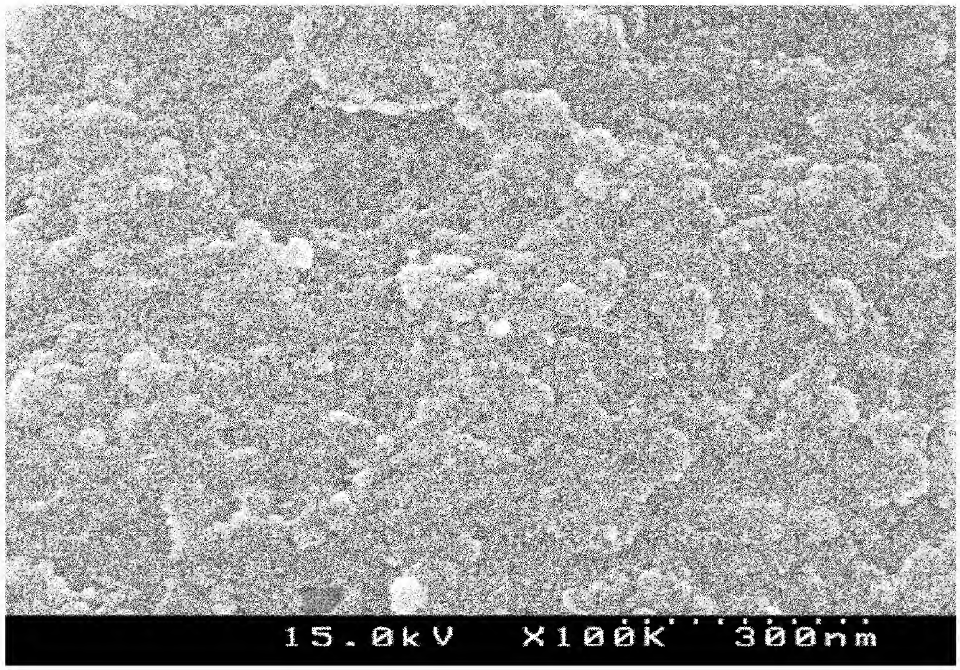

CPS
1560
1480
1400
1320
1240
1160
1080
1000
920
840
760
680
600
520
440
360
280
200
120
40
5.0
10.0
15.0
20.0
25.0
30.0
35.0
40.0
45.0
50.0
55.0
60.0
65.0
70.0
75.0
80.0
85.0
Deg.

FIG. 3



INTERNATIONAL SEARCH REPORT

International application No.
PCT/KR2004/001813

A. CLASSIFICATION OF SUBJECT MATTER

IPC7 B29C 71/00

According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)

IPC 7 B29C 71/00, H05B 3/00

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Korean Patents and applications for inventions since 1975
Korean Utility models and applictions for Utility models since 1975
Japanese Utility models and application for Utility models since 1975

Electronic data base consulted during the intertnational search (name of data base and, where practicable, search terms used)

eKIPASS, E-SPACENET

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
A	US 6,464,918 B1 (Heraeus Noblelight GmbH) 15 OCTOBER 2002 See the whole document	1, 11, 12, 16
A	WO 94/28693 A1 (EA TECHNOLOGY Lmtd.) 8 DECEMBER 1994 See abstract, claim 1	1, 11, 12, 16
A	KR 10-394981 B1 (OSAKA Prefectural Govennent) 4 AUGUST 2003 See claims 1-3	1, 12
A	JP 10-55877 A (ATSUSHI Ehata) 24 FEBRUARY 1998 See the whole document	1, 12

D Further documents are listed in the continuation of Box C.

☒ See patent family annex.

* Special categories of cited documents:
"A" document defining the general state of the art which is not considered to be of particular relevance
"E" earlier application or patent but published on or after the international filing date
"L" document which may throw doubts on priority claim(s) or which is cited to establish the publication date of citation or other special reason (as specified)
"O" document referring to an oral disclosure, use, exhibition or other means
"P" document published prior to the international filing date but later than the priority date claimed

"T" later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention
"X" document of particular relevance; the claimed invention cannot be considered novel or cannot be considered to involve an inventive step when the document is taken alone
"Y" document of particular relevance; the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art
"&" document member of the same patent family

Date of the actual completion of the international search	Date of mailing of the international search report
14 APRIL 2005 (14.04.2005)	15 APRIL 2005 (15.04.2005)

Name and mailing address of the ISA/KR

Korean Intellectual Property Office
920 Dunsan-dong, Seo-gu, Daejeon 302-701,
Republic of Korea

Facsimile No. 82-42-472-7140

Authorized officer

KIM, Hee Seung

Telephone No. 82-42-481-8157

INTERNATIONAL SEARCH REPORT
Information on patent family members

International application No.
PCT/KR2004/001813

Patent document cited in search report	Publication date	Patent family member(s)	Publication date
US 6,464,918 B1	15. 10. 02	EP 987923 A1	22. 03. 00
		JP 12-77166 A	14. 03. 00
WO 94/28693 A1	08. 12. 94	DE 69417231 01	22. 04. 99
		EP 700629 A1	13. 03. 96
		US 6057532 A	02. 05. 00
KR 10-394981 B1	04. 08. 03	EP 1076474 A1	14. 02. 01
		JP 3543174 B2	14. 07. 04
		US 6501056 B1	31. 12. 02
JP 10-55877 A	24. 02. 98	NONE	